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Grind under Reduced conditions

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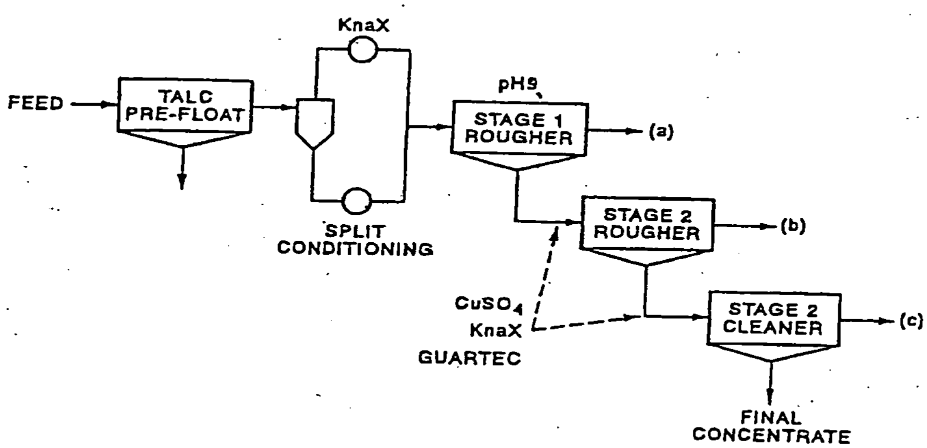

(57) Abstract

This invention relates to a process for the treatment of a sulphide ore containing pentlandite, pyrrhotite and possibly talc. The ore is first ground preferably under substantially non-reducing conditions and then the ground ore is subjected to a talc pre-float wherein fast floating talc can be recovered in a preliminary stage of flotation without an excessive loss of nickel. The sulphide-containing tailing from the talc pre-float is then subject to split conditioning followed by flotation in the absence of copper sulphate so as to selectively float pentlandite from pyrrhotite. The pentlandite float product may then be subjected to a conventional sulphide flotation treatment in the presence of an activator such as copper sulphate to selectively float the sulphides in the concentrates.

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PROCESSING OF ORES

Technical Field

This invention relates to the processing of nickel deposits, particularly those deposits where the nickel occurs predominantly in the sulphide mineral pentlandite.

Background Art

Nickel occurs in a variety of minerals of which the most economically important is the sulphide mineral pentlandite $(Fe,Ni)_{9\pm x}S_8$. Deposits containing pentlandite usually also contain other sulphides such as pyrrhotite $(Fe_{1-x}S)$, chalcopyrite $(CuFeS_2)$ and pyrite (FeS_2) and various non-sulphides including magnesium bearing silicates such as talc.

The processing of sulphide nickel deposits currently involves grinding to liberate the nickel sulphides followed by flotation in which gangue minerals are rejected. It is particularly important to reject nearly all of the minerals that contain magnesium because the more MgO there is in a nickel concentrate the higher the temperature for subsequent smelting, owing to the effect which magnesia has on slag viscosity. Any increase in smelting temperature increases energy costs and reduces the life of furnace refractories.

The approach usually used in flotation is to arrange the chemical conditions to be as favourable as possible for the flotation of nickel sulphides and as unfavourable as possible for recovery of magnesium bearing minerals, particularly those minerals such as talc which are naturally strongly floatable. For example, copper sulphate is often used to increase the rate of flotation

of nickel minerals while polysaccharides such as starches or guar gums are added to suppress the floatability of talc. This strategy produces a highly efficient separation of nickel from non-sulphide minerals with a minimum concomitant loss of nickel. However, there is almost no selective rejection of those sulphides that contain little or no nickel.

Until recently there was little incentive to reject gangue sulphides such as pyrrhotite. Indeed, the presence of such sulphides in concentrates was often considered desirable because of the small amount of nickel they sometimes contain and because their reaction with oxygen produces a great deal of heat and less fuel is therefore needed for smelting. The only real penalty for recovering the gangue sulphides was the cost of transporting them to the smelter. That the smelting of such concentrates produced large amounts of sulphur dioxide was rarely considered a real problem.

Over the last few years this situation has changed dramatically. High sulphur dioxide emissions are now no longer environmentally acceptable and more and more stringent restrictions are being imposed on the amount of sulphur that smelters can emit. As a result, much research has been directed towards developing a flotation method that allows the selective rejection of gangue sulphides and of magnesium bearing minerals. By rejecting as much of the gangue sulphides as possible before smelting, nickel production for a given emission of sulphur can be maximised.

Disclosure of the Invention

An object of the present invention is to provide a flotation process for the selective rejection of

pyrrhotite and optionally for the selective rejection of magnesium bearing minerals from nickel sulphide ores with minimum concomitant loss of nickel recovery.

Accordingly, in one aspect, the present invention provides a method for effecting the concentration of pentlandite from a sulphide ore containing pentlandite and pyrrhotite, the method comprising the steps of:

grinding the sulphide ore under substantially non-reducing conditions;

forming a pulp comprising the ground ore and a collector for pentlandite; and

subjecting the pulp to froth flotation to form a froth containing pentlandite.

We have found that interactions occur between sulphides as soon as the minerals begin to oxidise and that the chemistry of the grinding environment can make or mar any subsequent flotation separation. In particular, we have found that grinding some ores in a mill constructed of stainless steel rather than in a mill constructed of mild steel improves the subsequent separation of pentlandite from pyrrhotite without any real change in the separation from non-sulphide minerals.

The improvement is not the result of any change in particle size distribution, but rather is a consequence of a change in the grinding chemistry which greatly reduces the floatability of pyrrhotite but not that of pentlandite.

Stainless steel is far more resistant to corrosion than is mild steel and conditions in mills made of stainless steel are much more oxidising that those in mild steel mills. As a consequence, it is the sulphides rather than the grinding media that oxidise in a stainless steel mill. Mineral-mineral interactions are therefore greatly

enhanced compared to those in a mild steel mill and we presume that some such interaction is the reason pyrrhotite floats less strongly.

Where it is not practical to use stainless steels in industrial grinding mills, steels that are not highly reactive may be used. An oxygen containing gas such as air may be supplied during grinding to ensure that conditions are not strongly reducing. A mild steel mill with mild steel grinding media and having ports permitting access of air during grinding may be used. A rubber lined mill charged with corrosion resistant steel grinding media could be used.

The pH of most nickel flotation pulps ranges from slightly acidic to slightly alkaline and at such pH values sulphides usually float rapidly with collector alone in the absence of any interference from minerals such as talc. The rate of flotation of the sulphides can be increased still further by adding copper sulphate before the collector but once copper sulphate is added it is not possible to select between pentlandite and pyrrhotite.

If instead of adding copper sulphate, alkali is added to raise the pH to a value of not less than about 9, pyrrhotite floats much less strongly and there is some decrease in the recovery of pentlandite. By analysing products from tests done under such conditions we have discovered that nearly all of the nickel that is lost is of a size greater than 75 µm while the decrease in recovery of pyrrhotite results mostly from a decrease in the rate of flotation of particles of intermediate size (10 to 75 µm). Recognising this, we have applied a technique known as split conditioning, which was developed earlier in our laboratory (Anthony, Kelsall and Trahar,

1975; Trahar, 1981), to optimise the chemical conditions for different sizes of particles in the pulp.

Accordingly in a second aspect the present invention provides a process for the concentration of pentlandite from a sulphide ore containing pentlandite and pyrrhotite, the process comprising the steps of:

grinding the ore to an appropriate size range and forming a pulp of the ground ore;

separating the pulped ore into a relatively coarse fraction and a relatively fine fraction;

contacting each fraction with a collector, the major proportion of the collector being added to the coarse fraction and thereafter combining the fractions to form a collector treated pulp;

adjusting the pH of the pulp to a value not less than about 8, more preferably about 9; and

subjecting the pulp to froth flotation to produce a froth product containing pentlandite.

The adjustment of the pH to a value greater than about 9 may occur before or after treating the pulp with the collector.

Using the method of the second aspect of the invention we have achieved nickel recoveries similar to those achieved when copper sulphate is used but with much better rejection of pyrrhotite.

Preferably the ground ore should have an 80 percent passing size less than about 250μm. For ores that are finely disseminated, however, finer grinding or re-grinding of selected streams would be necessary to liberate the pentlandite for flotation.

Preferably the relatively coarse fraction has a size greater than about 75 µm.

The pulped ore may contain about 20% to 65% solids.

While many types of alkalis and collectors may be used, soda ash is particularly preferred as the pH modifier and the preferred collector is n-amyl xanthate. Sodium carbonate or lime may also be used as the pH modifier however lime may lead to some loss of selectivity between the sulphides. A mixture of alkalis and of collectors may be used. The water used in this step should have little dissolved copper.

The amount of collector and the proportion of the collector added to the coarse fraction need to be determined for each ore, but generally speaking more than half of the collector should preferably be added to the coarse fraction. In our laboratory tests three quarters of the collector was added to the coarse fraction and the other quarter to the fine fraction. This proportion may be varied but it is important that most of the collector is added to the coarse size.

The split size for conditioning and the conditioning time need to determined for each particular ore. In our tests, pulps were wet screened over a 75 µm sieve and each size fraction was conditioned with collector for 5 minutes. In practice a cyclone or a cylinder-cyclone could be used to classify the pulp. Two stages of classification might also be used to ensure a precise size split. A few minutes of conditioning would probably be sufficient in most cases.

A gangue depressant can also be added either during or after split conditioning but such a reagent must be used

judiciously because some nickel can be lost if too much is added.

A talc depressant such as guar gum may be used, again judiciously. It has been found that the addition of small amounts of Guartec diminishes the extent to which talc slows down the rate of pentlandite flotation. Preferably most of the talc depressant, if used, is added to the fine fraction

Any suitable frother may be used in the flotation step, for example Propylene Glycol. The type and amount of frother if any, need to be determined for each ore. For some ores, a frother might not be needed at all, or the plant water available at the site may produce strong frothing obviating the need to use a frothing agent.

The rate of pentlandite flotation after split conditioning is not quite as rapid as that in the presence of copper sulphate and collector and a longer time is required for flotation. In laboratory tests, flotation times have been up to 16 minutes.

A further rejection of non-sulphides in a second stage may be necessary before the concentrate produced by the process of the second aspect is acceptable for smelting. This can be accomplished readily using a method which is in principle very similar to that used in conventional processing. By adding an activator such as copper sulphate and a collector to the concentrate produced by the process in accordance with the second aspect, the sulphides can be floated very rapidly while the floatability of the non-sulphides can be suppressed using a suitable depressant. By including such a stage, concentrates assaying only about 2 percent MgO can be produced without much additional loss of nickel.

Smelters usually require that concentrates assay less than about 4 percent MgO.

Thus in yet a third aspect the invention provides a process in accordance with the second aspect comprising the further step of treating the concentrate produced by process of the second aspect with a collector for sulphides, an activator and optionally a depressant for non-sulphides and subjecting the treated product to froth flotation to produce a sulphide-containing froth.

The activator may be copper sulphate. We emphasise here again that while copper sulphate increases the rate of flotation of pentlandite, once it has been added no further selection between pentlandite and pyrrhotite is possible. This is why copper sulphate is not used until after much of the pyrrhotite has been rejected. The depressent may be a talc depressant such as guargum.

The amounts of copper sulphate, collector and talc depressant needed are much the same as for conventional processing. In laboratory tests, we have achieved excellent results using 250 g/t of copper sulphate, 150 g/t of amyl xanthate and 550 g/t of the talc depressant Guartec. Other collectors and gangue depressants may also be used. Lowering the pH for the second stage might be advantageous in some circumstances. Sulphuric acid may be a suitable acid to use.

Particularly good results can be achieved by operating the second stage in a rougher-cleaner configuration. It is important to minimise the recovery of water in cleaning because rejection of the non-sulphides is ultimately limited by entrainment.

Pneumatic cells such as column cells are reported to give lower water recoveries and to be particularly efficient for cleaning and might therefore be better suited than conventional cells for the cleaning of concentrates.

We have also found that the presence of large amounts of talc in nickel ores significantly reduces the rate of flotation of the sulphides, particularly when copper sulphate is not added. This problem can be overcome by allowing the talc to float without collector before sulphide flotation. Talc is naturally strongly floatable and the only reagent needed for such a pre-float is a frother. Frothers that give brittle froths, such as methylisobutylcarbinol (MIBC), are preferred. It may even be possible to pre-float without a frother.

Thus in yet a fourth aspect the present invention provides a process for the recovery of pentlandite from a sulphide ore containing pentlandite and pyrrhotite wherein the ore is comminuted and subjected to froth flotation in the absence of a collector to produce a talc-containing froth and a sulphide-containing tailing and thereafter subjecting the tailing to one or more sulphide flotation step.

The sulphide-containing tailing produced by the process of the fourth aspect may be used as the feed for the processes of the invention in its first, second and third aspects.

A small amount of nickel is recovered to the talc concentrate owing to the non-selective contribution which entrainment makes to the recovery of all minerals. However, this loss can be kept to a minimum by cleaning the talc concentrate and by minimising the amount of water recovered during flotation. Control of water recovery may

be achieved by keeping pulp level and aeration rate low in the cleaning step. Additionally the use of froth modifiers such as tri-butyl phosphate may be considered although as such modifiers can sometimes adversely affect flotation, they should be tested beforehand in a laboratory preferably using talc from the plant.

Column cells might again be suited more than conventional cells for cleaning.

A second option for handling the talc concentrate is to recycle it to a final stage which is in accordance with the fourth aspect of the present invention where sulphides are floated from talc. Recycling the concentrate in such a manner would provide an opportunity for any floatable nickel to be removed whilst ensuring that the feed to the pyrrhotite rejection stage contains little fast floating talc.

A third option is to reject part of the talc concentrate and recycle the rest in the manner just described. In this instance treatment is by reverse flotation of cleaned talc concentrate so as to produce a talc product-unfloated fraction-that can be rejected and a sulphide concentrate that can be recycled to the final stage; reagents, and minerals contacted with reagents must be removed from the pre-float circuit. The activator, collector and depressant for non-sulphides used in the final stage may be added in the reverse cleaner instead of being added to the final stage.

For ores that contain small amounts of talc or that contain only poorly floatable non-sulphides a pre-float might not be necessary; the floatability of such minerals might be suppressed readily by adding a suitable depressant during sulphide flotation.

In a preferred aspect the ore subjected to the process in accordance with the invention is ground and/or classified so as to minimise particles of a size less than about 10μm.

An important finding from our research is that poor floatability of fine (-10 μm) nickel places a very real limitation on the nickel recovery ultimately attainable. This limitation exists in our new process (more than half of the nickel we fail to recover in our laboratory tests is less than 10 μm in size) just as it does in standard laboratory tests using convention methods and in all of the plants for which we have recovery-size data.

We recognise the limitation imposed by the difficulty of selectively recovering fine nickel and seek to handle the problem by excellence in both grinding and classification. By efficient and precise grinding and classification and by careful regrinding of selected size fractions of selected streams the amount of nickel ground to very fine sizes can be minimised.

To apply the present invention most efficiently the particle size dependence of flotation should be known for each of the ores to be treated. This size dependence can be established by careful sizing of flotation products. Data from such sizings allow the optimum size range for flotation to be determined (see, for example Figure 5).

Brief Description of the Drawings

Figure 1 is a general flowsheet showing one arrangement of steps in a process in accordance with the invention

Figure 2 is a general flowsheet showing a second arrangement of steps in a process in accordance with the invention.

Figure 3 is a general flowsheet showing a third arrangement of steps in a process in accordance with the invention.

Figure 4 is a general flowsheet showing a third arrangement of steps in a process in accordance with the invention.

Figure 5 is a graph showing recovery-size dependance for one ore using a flotation method in accordance with the invention. N/P/S is a measure of pyrrhotite behaviour and NSG is a measure of the behaviour of all the non-sulphide gangue minerals including talc.

Figure 6 is a graph showing Nickel- N/P/S selectivity curves (ratio of average constants) for flotation of an ore ground in an open mild steel mill (test 1) and in a closed stainless stell mill (test 2).

Figure 7 is a graph showing Nickel-MgO selectivity curves (ratio of average rate constants) for flotation of an ore ground in an open mild steel mill (test 1) and in a closed steel mill (test 2).

Modes for Using the Invention

A scheme including the talc pre-float and the first sulphide flotation stage of the second aspect of the invention is shown in Figure 1.

Referring to Figure 1, the feed ore, which has preferably been ground under substantially non-reducing conditions,

is suppli d to a talc pre-float rougher which may be, for example, a series of Agitair cells. In this particular arrangement, the talc recovered from the pre-float is discarded whilst the sulphide-containing tailings are subjected to split conditioning, that is, the sulphide-containing tailing is separated into a relatively coarse fraction and relatively fine fraction and both fractions are conditioned with potassium n-amyl xanthate, the major proportion of the xanthate being used to treat the coarse fraction, and then the two conditioned fractions are recombined.

The conditioned sulphide concentrate is treated in two stages, stage 1 being the first stage of sulphide flotation and stage 2 the second. The conditioned sulphide concentrate is first treated in a stage 1 rougher at pH 9 in the absence of copper sulphate to selectively reject some of the pyrrhotite and recover a pentlandite-containing concentrate. The pentlandite-containing concentrate is then subjected to conventional sulphide flotation in a second stage rougher-cleaner arrangement in the presence of copper sulphate, potassium n-amyl xanthate and a talc depressant such as guartec to produce a final nickel concentrate and tailing streams which are rejected.

In practice, the tailings from both sulphide flotation stages (streams a, b and c in Figure 1) would be re-cycled or subject to further treatment so as to maximise nickel recovery. The most appropriate place to recycle such streams and the best methods of further treatment need to be determined on a case-by-case basis. For example, for ores that are difficult to grind or which are finely disseminated much of the pentlandite in the tailings might be locked with other minerals. Regrinding of composite

particles would therefore be necessary before a further stage of flotation to recover the nickel.

Figure 2 illustrates an alternative arrangement of stages in which the talc-containing concentrate floated in the pre-float rougher is subjected to cleaning in a pre-float cleaner and where the tailings are returned to the pre-float rougher and the talc rejected.

Figure 3 illustrates another possible arrangement wherein the talc-containing concentrate from the talc pre-float rougher is combined with the sulphide-containing concentrate floated from the stage 1 rougher.

Figure 4 illustrates a further possible arrangement in accordance with the invention wherein part of the talc concentrate is rejected in a "reverse cleaner" and the rest is recyced to the final stage of the process where talc is rejected from the sulphides.

In order that the invention might be understood more readily the following non-limiting examples are provided.

Example 1 Effect on Sulphide Flotation Stage 1 Results of Grinding in Mills Constructed of Different Steels

The sample used was from a nickel deposit containing 2.38% Ni, 14.5% MgO, 6.79% S and 13.5% Fe. Mineralogical analyses indicated that the nickel occurred almost exclusively as pentlandite and that pyrrhotite was the predominant sulphide gangue mineral. The principal magnesium bearing minerals were talc, magnesite and silicates of the chlorite group. Microprobe analyses

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showed that none of the gangue minerals contained more than about 0.5% nickel and that most contained much less.

Laboratory ball mills constructed of either stainless steel or of mild steel with media of similar type were used to grind the ore. Changing from one mill to the other produced little change in the particle size distribution after grinding. Products from grinding were 80 percent by weight passing about 75 µm. The ore was ground in 500 gram lots at 67 percent solids using distilled water.

A modified Denver laboratory cell was used for flotation. Owing to the presence of talc, tests included a 2 minute pre-float in which the only reagent added was a frother. The tailing from this pre-float was then wet-screened over a 75 µm sieve and the two size fractions separately conditioned with collector before flotation.

Either potassium n-amyl xanthate (KnaX) or potassium ethyl xanthate (KeX) was used as collector. Three quarters of the collector was added to the sieve over-size and the remaining quarter was added to the under-size.

The frother was polypropylene glycol (Cyanamid Aerofroth 65) made up as a 0.25 percent solution. An initial dose of 2ml of frother solution was added before the talc pre-float and thereafter 2ml per minute was added from an automatic pump to maintain an active froth column.

The flotation gas was bottled air (a synthetic mixture of O_2 and N_2) and was supplied at a flow rate of 8 litres/minute. The pH was adjusted to 9 and controlled automatically at this value using a dilute sodium hydroxide solution. All water was distilled.

Concentrates and tailings were sampled in a standard manner and assayed for Ni, S and MgO.

Results from the tests are given in Table 1. In this table the letters A and R refer to the cumulative concentrate assay and to the cumulative recovery of each species named. These values include losses to the talc pre-float. The behaviour of the non-pentlandite sulphur (N/P/S) minerals was calculated assuming that pentlandite contains 33.0 percent nickel and 33.0 percent sulphur. As has already been indicated, pyrrhotite was the predominant sulphide gangue mineral and it is therefore assumed that non-pentlandite sulphur is mostly pyrrhotitic sulphur although it is known that the ore contains some pyrite.

Comparing tests 1 and 2 in Table 1 shows that the pyrrhotite floated less strongly from ore ground in the stainless steel mill than from ore ground in the mild steel mill after the addition of n-amyl xanthate. The change in floatability was substantial with the N/P/S recovery being lowered by more than 13 percent. When ethyl xanthate rather than n-amyl xanthate was added, the pyrrhotite again floated less strongly from ore ground in the stainless steel mill (compare tests 3 and 4 in Table 1).

The improvement in the selectivity of separation of nickel from pyrrhotite when the stainless steel mill is substituted for the mild steel mill can be seen clearly when the rate data for test 1 (mild steel mill grind) and for test 2 (stainless steel mill grind) are plotted in the form of selectivity curves (Figure 6). Assuming first order kinetics, the slope of the lines is the ratio of the average rate constants for the flotation of the nickel mineral and for the N/P/S minerals. A line of 45^{o} indicates no selectivity; a line down the ordinate (the

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vertical axis) indicates perfect selectivity between Ni and N/P/S.

Plotting nickel-MgO selectivity curves for the same tests (Figure 7) shows that there is little or no change in the selectivity of separation of pentlandite from magnesium bearing minerals.

Table 1 - Stage 1 Sulphide Flotation Results after Grinding in a Steel Mill and after Grinding in a Stainless Steel Mill.

			Component				
Test	Description		Ni	N/P/S	MgO	NSG	H_2O
1	30 mg KnaX +75μm 10 mg KnaX -75μm Mild Steel Mill 16 minutes flotation pH 9	A R	4.91 91.0	5.83 67.4	15.7 48.6	70.4 39.7	 80.8
2	30 mg KnaX +75μm 10 mg KnaX -75μm Stainless Steel Mill 16 minutes flotation pH 9	A R	5.15 89.9	5.47 54.1	15.7 45.4	70.6 36.0	 74.7
3	30 mg KeX +75μm 10 mg KeX -75μm Mild Steel Mill 8 minutes flotation pH 9	A R	5.74 83.1	7.11 54.0	15.5 37.7	64.6 27.1	 49.5
4	30 mg KeX +75μm 10 mg KeX -75μm Stainless Steel Mill 8 minutes flotation pH 9	A R	5.98 81.3	6.34 46.3	15.6 36.7	65.8 26.4	 46.9

Example 2 Split Conditioning

The floatability of coarse nickel at pH 9 can be raised by using split conditioning. In this example the collector was divided between two parallel conditioning stages and flotation carried out for 16 minutes. The split conditioning approach was compared with the conventional process using copper sulphate and ethyl xanthate and the results are shown in table 2.

Table 2 A comparison between the Efficiency of Split Conditioning and of adding $CuSO_4$ for the separation of Nickel from N/P/S (16 min results)

			Ni	N/P/S	MgO	N/S/G	H_2O
37	pH 9 -100mg $CuSO_4$	A	4.27	8.12	14.1	66.5	
	40mg KeX	R	91.8	88.4	50.1	40.3	81.1
35	pH 9 -30mg KeX +75µm	A	4.60	6.33	15.2	70.1	
	10mg KeX -75µm	R	89.2	64.4	49.4	39.4	81.7
27	pH 9- 30mg KnaX +75µm	A	4.91	5.83	15.7	70.4	
	10mg KnaX -75µm	R	91.0	67.4	48.6	39.7	80.8

It will be seen from Table 2 that for equivalent Nickel recovery the split conditioning examples (35 and 27) recover less pyrrhotite as compared to the the conventional method (37) for the same level of pentlandite recovery.

Example 3 Results from a Laboratory Test using the Invention.

The first test of Example 1 was repeated but this time a second stage of sulphide flotation was added to reject non-sulphides from the concentrate. This second stage included both roughing and cleaning. The flotation time for the rougher was 8 minutes and for cleaning the time of flotation was 6 minutes. Pulps were conditioned before both the rougher and the cleaner and the order of addition of reagents was as follows:

Rougher:

- pH of the pulp adjusted to 7 using sulphuric acid
- 100 mg of $CuSO_4 5H_2O$ conditioned for 2 minutes
- 20 mg of KnaX conditioned for 2 minutes
- 150 mg of Guartec conditioned for 2 minutes

Cleaner:

- 10 mg KnaX conditioned for 2 minutes
- 15 mg Guartec conditioned for 2 minutes

Additions of 20 mg of KnaX and 50 mg of Guartec were also made after 2 and 4 minutes of rougher flotation while additions of 10 mg of KnaX and 5 mg of Guartec were made after 2 and 4 minutes of cleaner flotation. Frother was added as required during both roughing and cleaning.

A particular effort was made to minimise entrainment in cleaning. To reduce the water recovery the pulp level was lowered, a minimum of frother was added and the froth scraping rate was decreased from every 5 seconds to every 10 seconds.

The results for each stage of the test are as follows:

			Component			
Stage	Description		Ni	N/P/S	MgO	NSG
Talc Pre-Float	Frother Only 2 minutes flotation	A	0.68	0.31	27.4	97.2
		R	2.42	0.60	16.0	9.92
Stage 1 Rougher	30 mg KnaX +75μm 10 mg KnaX -75μm 16 minutes flotation pH 9	A	4.95	6.03	15.2	69.8
		R	92.2	60.8	46.5	37.2
Stage 2 Rougher	100 mg $CuSO_4.5H_2O$ 50 mg KnaX 250 mg Guartec 8 minutes flotation pH 7	A	13.7	14.1	5.22	22.8
		R	83.7	46.8	5.23	3.99
Stage 2 Cleaner	25 mg $CuSO_4.5H_2O$ 25 mg KnaX 25 mg Guartec 6 minutes flotation pH 7	A	16.5	16.9	2.28	7.53
		R	81.0	44.9	1.84	1.06

Of the nickel originally in the sample, 81% was recovered to the final (stage 2 cleaner) concentrate at a grade of 16.5%. This is a particularly good result for this ore and by way of comparison, standard laboratory flotation methods produce concentrates assaying only about 9% nickel at 80% recovery.

It should also be recognised that:

(a) none of the tailings were recycled, that is, the nickel which reported to the rougher tailings and the cleaner tailings were treated as a loss, and

(b) we used only a single stage of grinding without classification. As a consequence about 20% of the nickel was ground to less than 10 μm in size and of this we recovered less than half.

With further treatment and with recycling of products it is not unreasonable to expect that the result can be improved still further, particularly when the limitations imposed by the poor floatability of the fine nickel are addressed as outlined in step 5 of our Disclosure of the Invention.

References

Anthony, R.M., Kelsall, D.F., and Trahar, W.J., 1975. The effect of particle size on the activation and flotation of sphalerite. Proc. Australas, Inst.Min.Metall., Vol.254, pp.47-58.

Trahar, W.J., 1981. A rational interpretation of the role of particle size in flotation. Int. J. Miner. Process., Vol.8, pp.289-327.

SUBSTITUTE SHEET

CLAIMS

1. A process for effecting the concentration of pentlandite from a sulphide ore containing pentlandite and pyrrhotite, the process comprising the steps of:

grinding the sulphide ore under substantially non-reducing conditions;

forming a pulp comprising the ground ore and a collector for pentlandite; and

subjecting the pulp to froth flotation to form a float product containing pentlandite.

2. A process in accordance with claim 1 wherein grinding is carried out in a mill wherein the grinding media is steel which is not highly reactive.

3. A process according to claim 2 wherein the steel is stainless steel.

4. A process according to claim 2 wherein the grinding is carried out in a rubber lined mill charged with corrosion resistant steel grinding media.

5. A process according to any one of claims 1 to 4 wherein grinding is carried out in the presence of an oxygen-containing gas.

6. A process according to claim 5 wherein the oxygen-containing gas is air.

7. A process according to claim 6 wherein the grinding media is mild steel.

8. A process for the concentration of pentlandite from a sulphide ore containing pentlandite and pyrrhotite, the process comprising the steps of:

grinding the ore to an appropriate size range to form a pulp of the ground ore;

separating the pulped ore into a relatively coarse fraction and a relatively fine fraction;

contacting each fraction with a collector, the major proportion of the collector being contacted with the coarse fraction and thereafter combining the fractions to form a collector treated pulp;

adjusting the pH of the collector treated pulp to not less than about 8; and

subjecting the pulp from the preceding step to froth flotation to produce a froth product containing pentlandite.

9. A process according to claim 8 wherein the pH of the collector treated pulp is adjusted to about 9.

10. A process according to claim 8 wherein pH adjustment occurs before treating the pulp with the collector.

11. A process according to claim 8 or 9 wherein the collector is a xanthate, preferably potassium n-amyl xanthate.

12. A process according to any one of claims 8 - 11 wherein the coarse fraction has a particle size greater than about 75µm.

13. A process according to any one of claims 8 - 12 wherein a gangue depressant is added before or after forming the collector treated pulp.

14. A process according to any one of claims 8 - 13 including the further step of treating the froth product with a collector for sulphides, an activator and optionally a depressant for non-sulphides and subjecting the treated product to froth flotation to produce a sulphide-containing froth.

15. A process according to claim 14 wherein the activator is copper sulphate.

16. A process according to claim 14 or 15 wherein the collector is a xanthate.

17. A process according to any one of claims 14-16 wherein the depressant is a depressant for talc.

18. A process according to any one of claims 8-17 wherein the ore is ground under substantially non-reducing conditions.

19. A process in accordance with claim 18 wherein grinding is carried out in a mill wherein the grinding media is steel which is not highly reactive.

20. A process according to claim 19 wherein the steel is stainless steel.

21. A process according to claim 19 wherein the grinding is carried out in a rubber lined mill charged with corrosion resistant steel grinding media.

22. A process according to any one of claims 18 to 21 wherein grinding is carried out in the presence of an oxygen-containing gas.

23. A process according to claim 22 wherein the oxygen-containing gas is air.

24. A process according to claim 23 wherein the grinding media is mild steel.

25. A process according to any one of claims 8 to 24 wherein the sulphide-containing ore is first subjected to a talc pre-float to remove talc.

26. A process according to claim 25 wherein the ore is comminuted and then subjected to froth flotation in the absence of a collector to produce a talc-containing froth concentrate and a sulphide-containing tailing.

27. A process according to claim 26 wherein the talc-containing concentrate is combined with the froth product of the further step of claim 14.

28. A process according to claim 27 wherein part of the talc-containing concentrate is combined with the sulphide-containing concentrate.

29. A process for the recovery of pentlandite from a sulphide ore containing pentlandite and pyrrhotite wherein the ore is comminuted and subjected to froth flotation in the absence of a collector to produce a talc-containing froth and a sulphide-containing tailing and thereafter subjecting the tailing to one or more sulphide flotation steps.

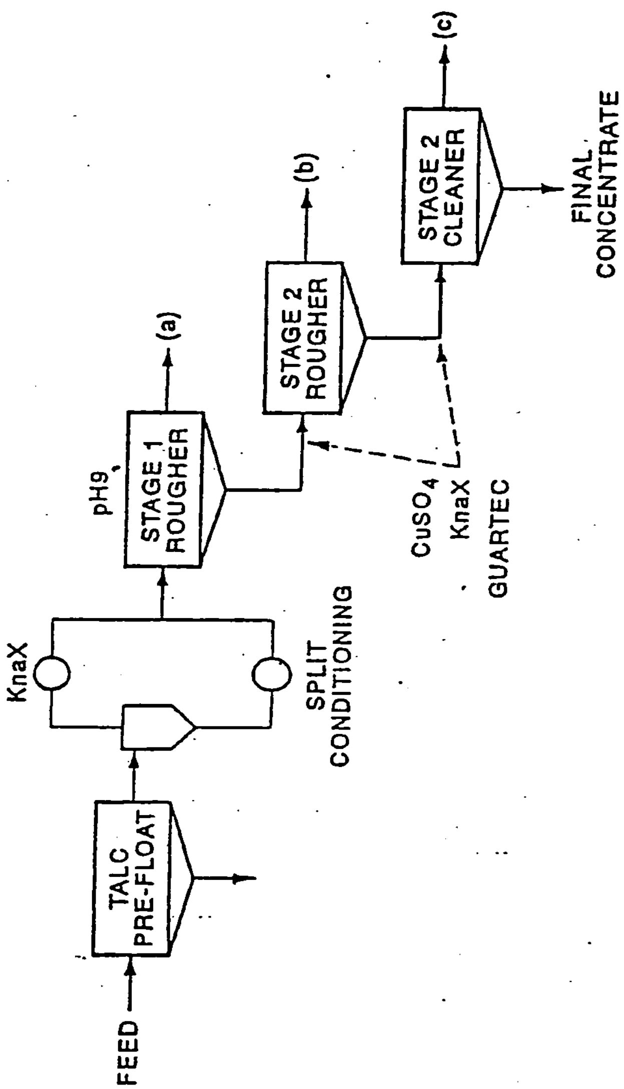
FEED
TALC PRE-FLOAT
KnaX
SPLIT CONDITIONING
pH9
STAGE 1 ROUGHER
(a)
STAGE 2 ROUGHER
(b)
$CuSO_4$
KnaX
GUARTEC
STAGE 2 CLEANER
(c)
FINAL CONCENTRATE

Figure 1.

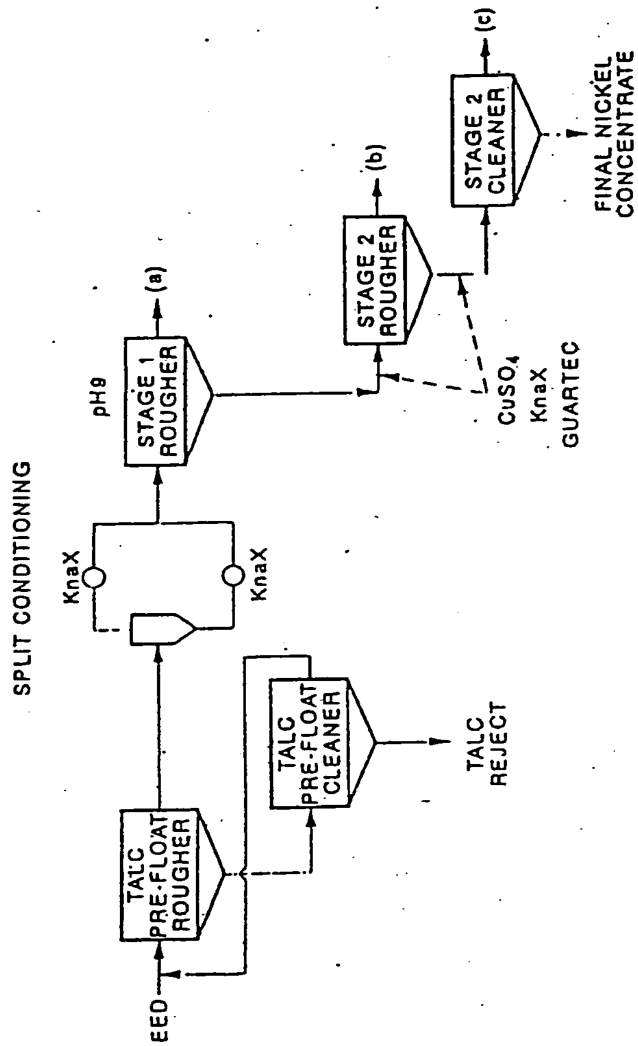

Figure 2.

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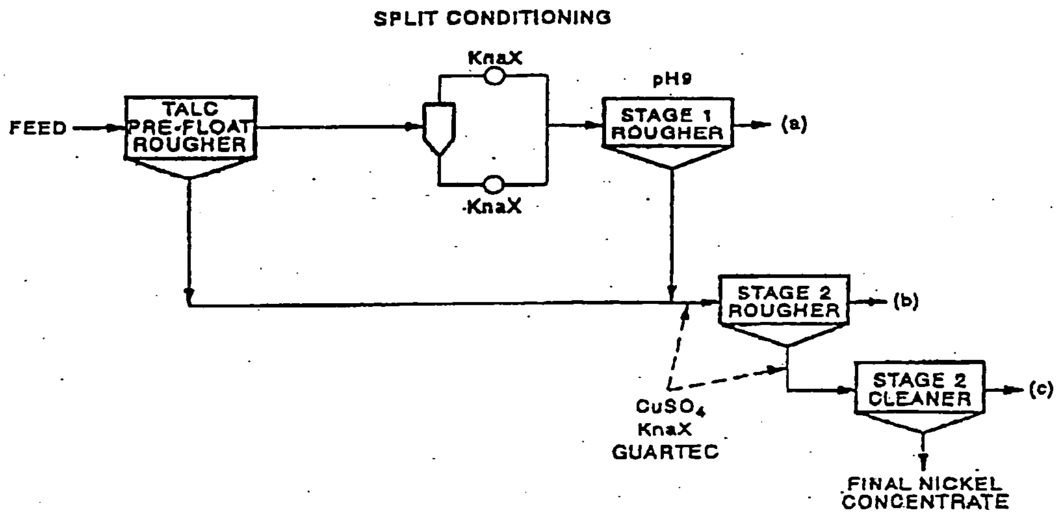
SPLIT CONDITIONING
KnaX
pH9
FEED
TALC
PRE-FLOAT
ROUGHER
STAGE 1
ROUGHER
(a)
KnaX
STAGE 2
ROUGHER
(b)
STAGE 2
CLEANER
(c)
$CuSO_4$
KnaX
GUARTEC
FINAL NICKEL
CONCENTRATE

Figure 3.

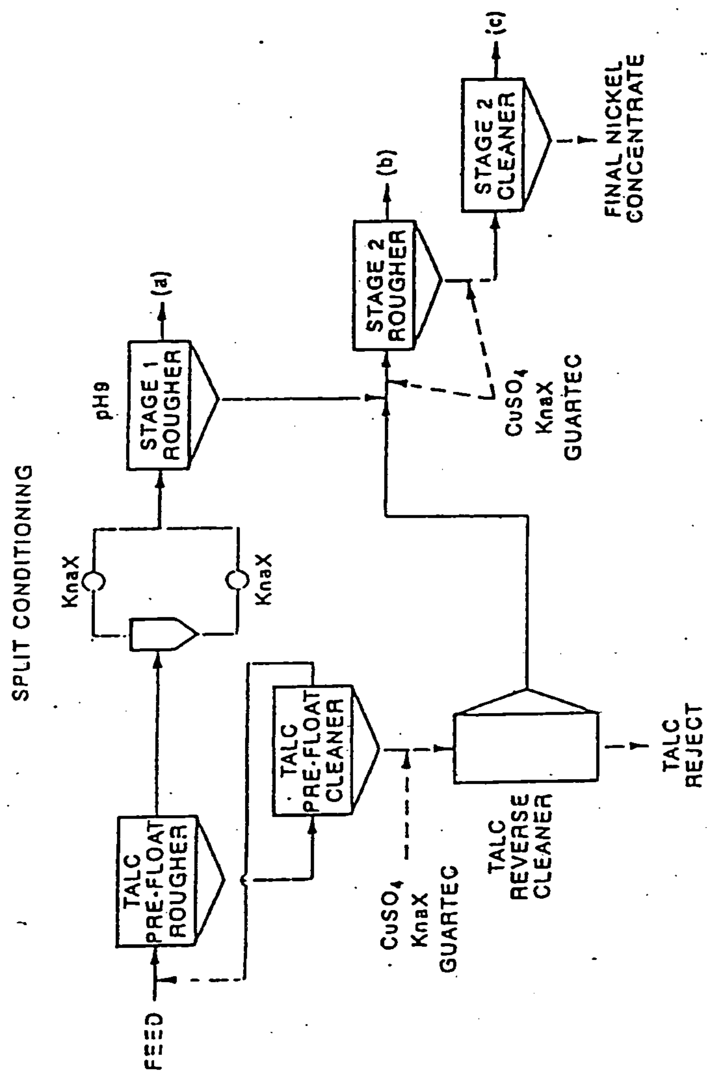

Figure 4.

SUBSTITUTE SHEET

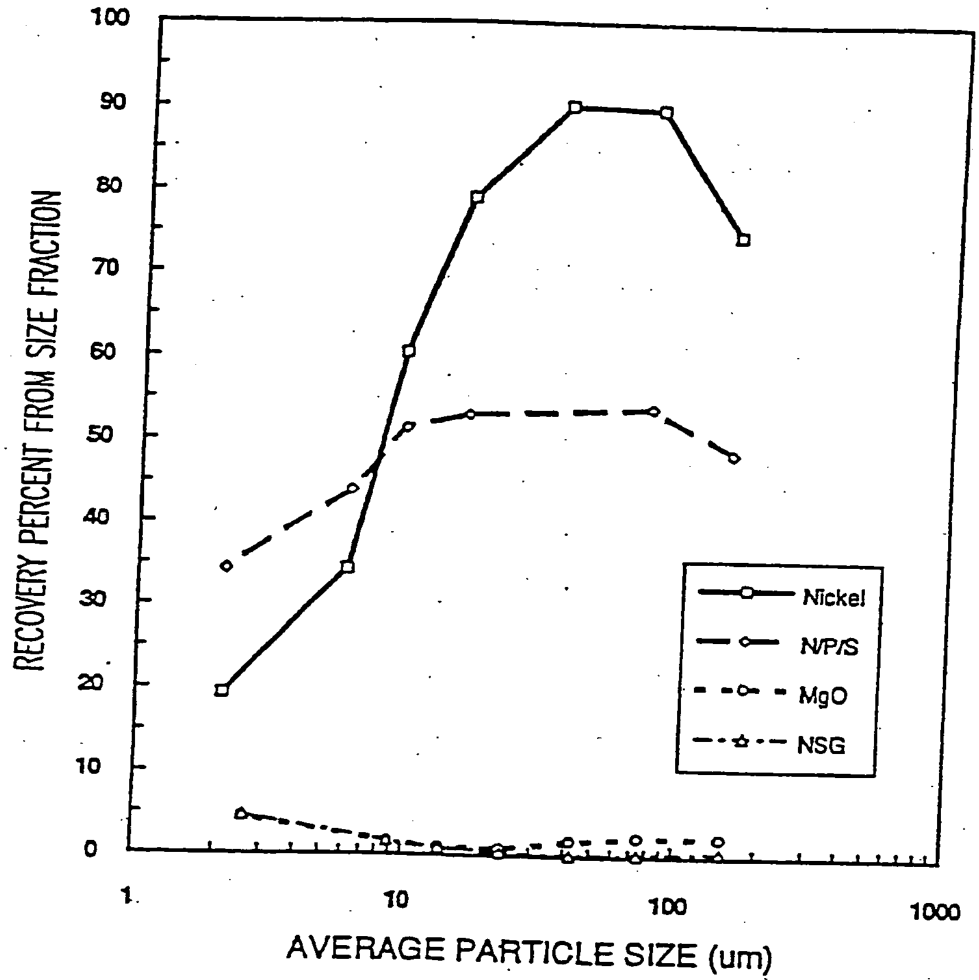

Figure 5.

SUBSTITUTE SHEET

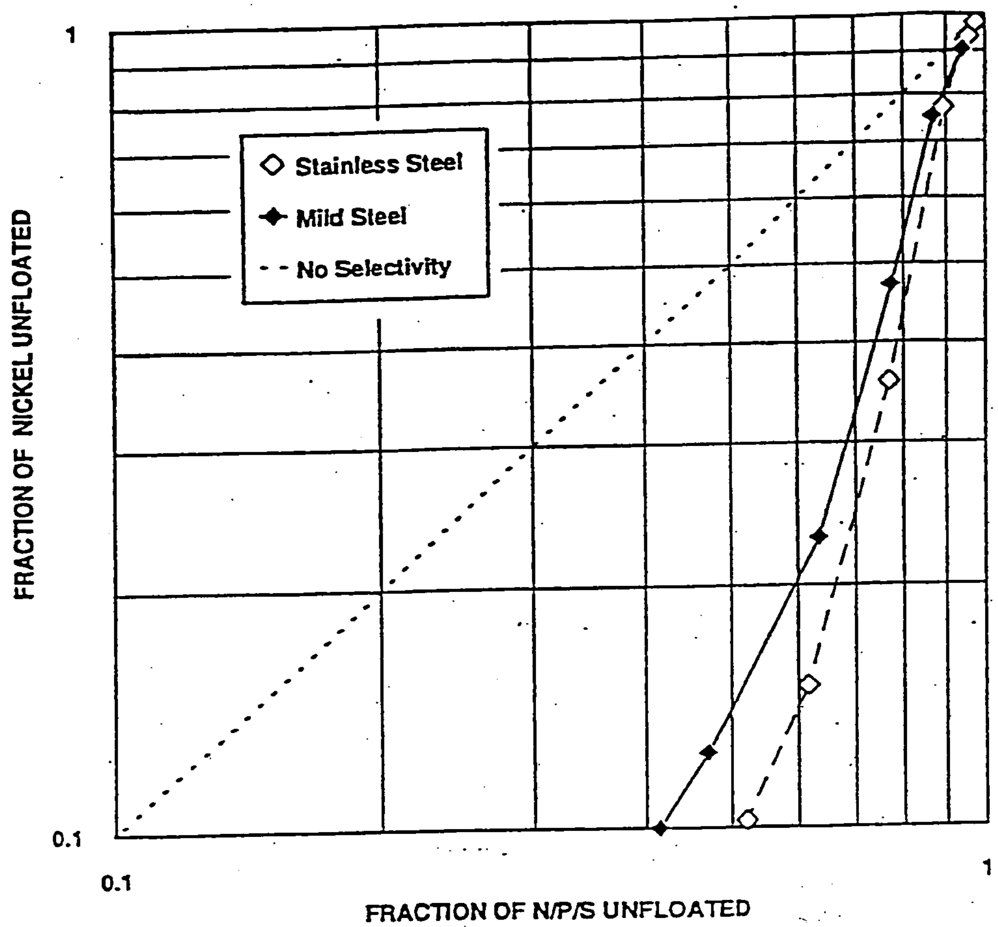

Figure 6.

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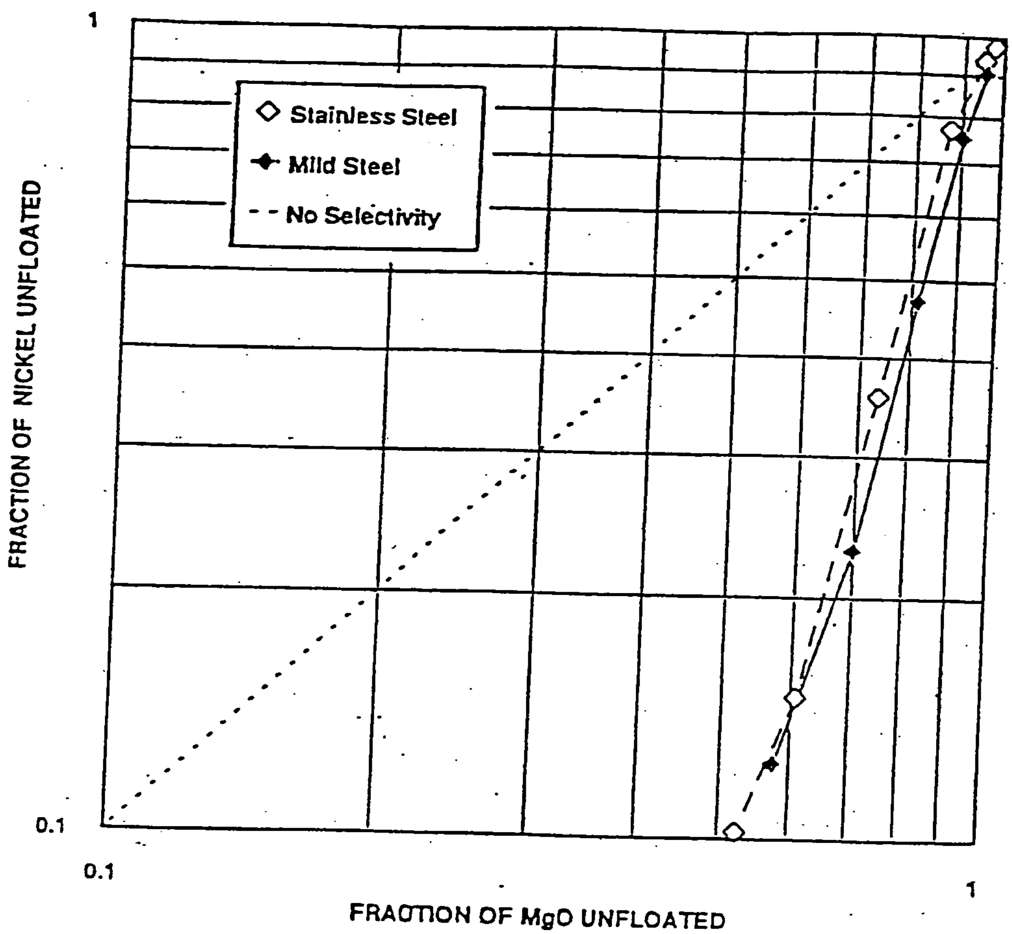

Figure 7.

SUBSTITUTE SHEET

INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU92/00450

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.[5] B03D 1/02 // C01G 53/11, 53/00

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC B03D 1/02

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
AU : IPC as above

Electronic data base consulted during the international search (name of data base, and where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
X	WO,A, 89/00457 (THE LUBRIZOL CORPORATION) 26 January 1989 (26.01.89) Whole document	(1-2,7)
X	US,A, 3309029 (FRAME) 14 March 1967 (14.03.67) See col 1 lines 1-25, col 2 lines 1-5	(1-2,7)
X	US,A, 4256227 (PETROVICH) 17 March 1981 (17.03.81) Whole document	(1-2,7)

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
"E" earlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
24 November 1992 (24.11.92)	4 Dec 1992 (04.12.92)

Name and mailing address of the ISA/AU	Authorized officer
AUSTRALIAN PATENT OFFICE PO BOX 200 WODEN ACT 2606 AUSTRALIA	B Bourke B. BOURKE
Facsimile No. 06 2853929	Telephone No. (06) 2832148

Form PCT/ISA/210 (continuation of first sheet (2)) (July 1992) copjne

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate of the relevant passages	Relevant to Claim No.
X	GB,A, 2086768 (INCO LIMITED) 19 May 1982 (19.05.82) Whole document	(1-2,7)
X	CA,A, 1104274 (INCO LIMITED) 30 June 1981 (30.06.81) Whole document	(1-2,7)
X	WO,A, 86/06983 (THE DOW CHEMICAL COMPANY) 4 December 1986 (04.12.86) Whole document	(1-2,7)
X	AU,A, 26603/71 (WESTERN MINING CORPORATION LIMITED) 21 September 1972 (21.09.72) See page 2 lines 1-2, page 4 lines 5-6,27 & page 5 lines 1-2	(1-4,7)

Box I **Observations where certain claims were found unsearchable (Continuation of Item 1 of first sheet)**

This international search report has not established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim Nos.:
because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II **Observations where unity of invention is lacking (Continuation of item 2 of first sheet)**

This International Searching Authority found multiple inventions in this international application, as follows:

See Attached Sheet

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims

2. ☒ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

☐ The additional search fees were accompanied by the applicant's protest.

☐ No protest accompanied the payment of additional search fees.

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Member					
WO	8900457	AU	20829/88	EP	324833	NZ	225376
		ZA	8805062	ZM	41/88	ZW	94/88
		US	4879022				
CA	1096514	AU	41510/78	PH	13634	ZA	7900069
GB	2086768	AU	68322/81	BR	8101620	CA	1156380
		FI	810851	JP	57001456	ZA	8101606
WO	8606983	AU	54564/86	BR	8606705	CA	1270076
		CN	86101682	ES	552027	FI	870368
		NO	870393	PH	23738	PL	257991
		SE	8700376	YU	232/86	ZA	8601169
		ZM	12/86	ZW	40/86	US	4732668
		US	4735711	JP	63100961		

END OF ANNEX

Box II OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING

A lack of unity of invention exists between claims 1-7,8 and 29. As the claims are defined, claim 1 is directed to a process for concentrating pentlandite from sulphide ore involving the steps of grinding under non-reducing conditions, pulping and froth flotation (with a collector). Claim 8 is directed to a process for pentlandite recovery involving grinding, pulping, separaring the pulp into fine and coarse fractions which are then individually treated with a collector. The fractions are then recombined, pH adjusted and then froth flotated. Claim 29 again is directed to a process for pentlandite recovery, including the steps of comminution, froth flotation using no collectors and one or more sulphide flotation steps. Claims 1-7 are clearly linked to one invention, having the inventive concept of a process having grinding under non-reducing conditions. However, claim 8 identifies its invention as a process involving a separation step where each fraction is individually treated with a collector before pH adjusted and froth flotation. Lastly, claim 29 defines its invention as a process that involves an initial froth flotation that uses no 'collector'. Therefore, when considering all independent claims, lack of unity exists, as claim 8 and 29 have no common inventive feature of claims 1-7.